IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the application of: Snow et al.
Serial No.: 09/544,344
Filed: 04/06/2000
For: MATERIALS, METHOD AND APPARATUS FOR DETECTION AND MONITORING OF CHEMICAL SPECIES
Examiner: Soderquist, Arlen
Art Group Unit: 1743

Honorable Commissioner of Patents
PO Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.131 OF ARTHUR W. SNOW

Sir:

I, Arthur W. Snow, hereby declare that:

1. I am a joint inventor of the invention claimed in the above-identified patent application ("application").

2. Attached is a copy of portions of my invention disclosure, which formed the basis of the application.

3. The page titled "Patent Disclosure" bears a disclosure date of 05/15/1997. The contents of the disclosure were complete as of that date.

4. Attached are copies of pages from my laboratory notebooks dated 01/08/1997 and from 05/14/1997 to 06/20/1997. To the best of my recollection, all my research performed in the course of my full-time employment from 05/14/1997 to 06/20/1997 is documented in these pages.

5. Conception of the use of a coupling agent is documented on the notebook page dated 01/08/1997.

6. During the period of from 05/14/1997 to 06/20/1997, all of my research was concerned with encapsulated clusters, either as claimed in the application or directly or indirectly related to the claimed subject matter.

7. The entries of 06/20/1997 document the actual reduction to practice of the use of a coupling agent.

8. All work described in the disclosure and the notebook pages was performed in NAFTA or WTO member countries.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

31 May 2007
Date

Arthur W. Snow

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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DECLARATION UNDER 37 C.F.R. § 1.131 OF HENRY WOHLTJEN

Sir:

I, Henry Wohltjen, hereby declare that:

1. I am a joint inventor of the invention claimed in the above-identified patent application ("application").

2. Attached is a copy of portions of my invention disclosure, which formed the basis of the application.

3. The page titled "Patent Disclosure" bears a disclosure date of 05/15/1997. The contents of the disclosure were complete as of that date.

4. Attached are copies of pages from the laboratory notebooks of co-inventor Arthur W. Snow dated 01/08/1997 and from 05/14/1997 to 06/20/1997.

5. Conception of the use of a coupling agent is documented on the notebook page dated 01/08/1997.

6. The entries of 06/20/1997 document the actual reduction to practice of the use of a coupling agent.

7. All work described in the disclosure and the notebook pages was performed in NAFTA or WTO member countries.

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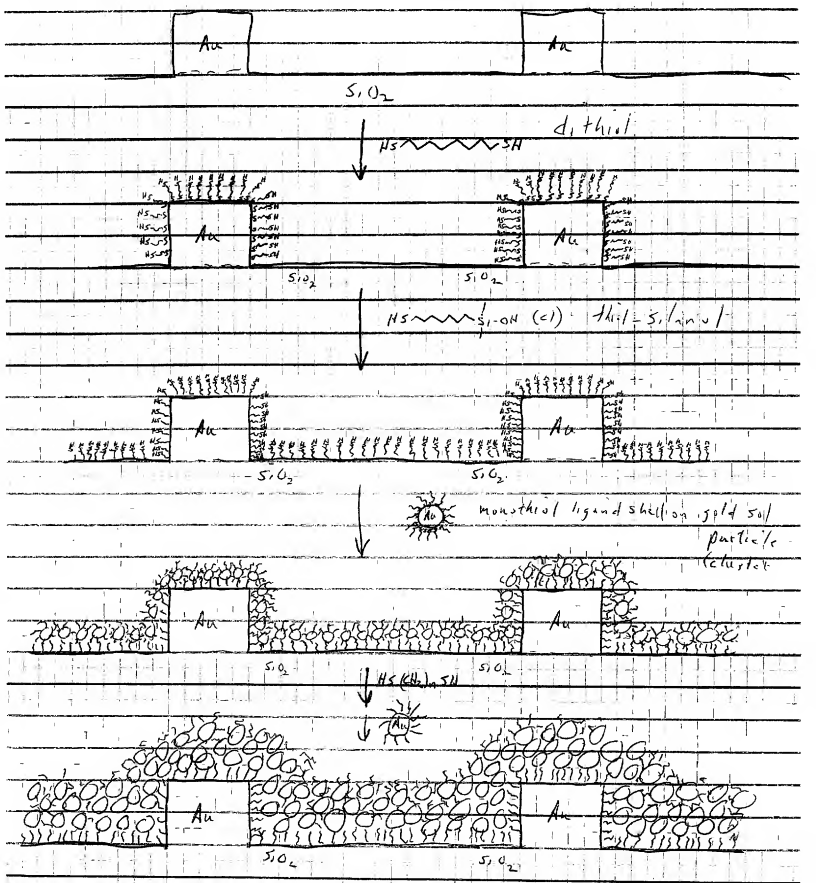
Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

30 May 2007

Date

Henry Wohlgen

Approach for Thiol-Gold self Assembly on Gold/Quartz IDE Transdu



d and understood (obtain two signatures):

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NAVAL RESEARCH LABORATORY

LABORATORY RECORD

BOOK No. N-7950

Assigned to Arthur W Snow

Dates 3-12-97 to 5-20-97

Surface Flow

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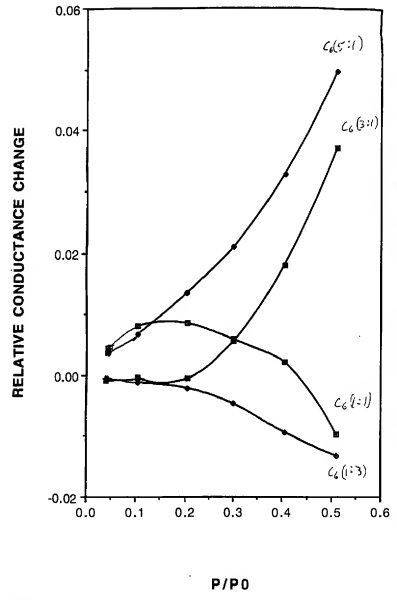

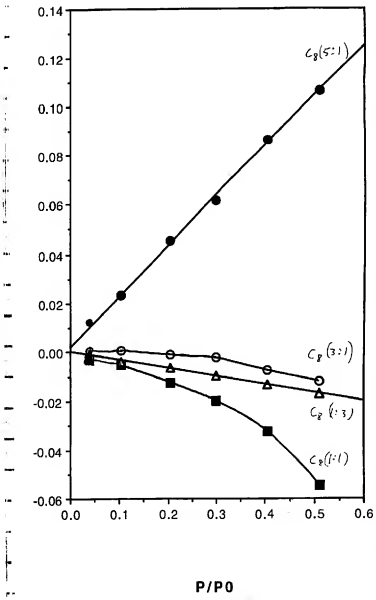

Trends appears to be as follows:

(1) Increasing chain length of ligand shell results in a decreasing and more negative response. The C_6 behavior is somewhat anomalous

(2) The C_{16}, C_{12} and C_8 chain length ligand shells displayed reasonably good linearity in their isotherms while the C_4 and C_6 were particularly non linear

(3) Large cluster cores promote positive sensor responses and small ones

Read and understood (obtain two signatures):

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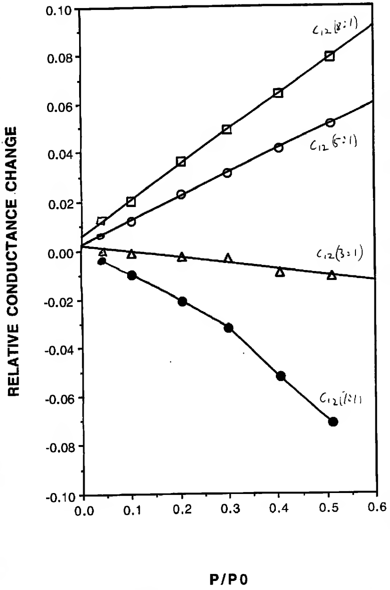

promote a negative response (C8 1:3 is an exception).

(4) C12 1:1 displays a crossover behavior (negative → positive response with decreasing vapor concentration).

Read and understood (obtain two signatures):

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Tetrachloroethylene Isotherm

Objective: Vapor Response mapping of $Au\ C_n(X:Y)$ to CCl_2CCl_2

5V/100KΩ Series	P/P°	Dilution Factor	Baseline	Signal	Signal/Baseline	Slope
Au C8 (3:1)						
	.512	$(88)^3$	171952	69816	.5940	1.16
	.405	$(74)^3$	175332	93254	.4681	1.16
	.301	$(62)^3$	175298	109216	.3770	1.25
	.205	$(58)^3$	176668	130178	.2631	1.28
	.104	$(47)^3$	176814	150680	.1478	1.42
	.051	$(37)^3$	177522	164724	.0721	1.42
	.00025	$(65)^3$	177672	Δ=55	.00031	2.48
Au C8 (1:1)						
	.512	$(80)^3$	21394	8419	.6065	1.18
	.405	$(68)^3$	21702	11192	.4843	1.20
	.301	$(62)^3$	21520	13224	.3855	1.28
	.205	$(59)^3$	21702	15790	.2724	1.33
	.104	$(47)^3$	21476	18246	.1564	1.45
	.051	$(37)^3$	21704	20080	.0748	1.48
	.00025	$(65)^3$	21506	Δ46	.00028	2.24
Au C6 (1:1)						
	.512	$(80)^3$	126970	51852	.5916	1.16
	.405	$(74)^3$	129878	67292	.4819	1.19
	.301	$(62)^3$	129514	78852	.3912	1.30
	.205	$(59)^3$	130468	93416	.2840	1.39
	.104	$(47)^3$	130172	109244	.1668	1.55
	.051	$(37)^3$	130610	119808	.0827	1.68
	.00025	$(65)^3$	129468	10835	.00027	2.16
Au C12 (8:1)						
	.512	$(80)^3$	71694	34332	.5211	1.02
	.405	$(74)^3$	70830	41858	.4090	1.01
	.301	$(62)^3$	70840	48378	.3171	1.05
	.205	$(59)^3$	70754	54960	.2232	1.09
	.104	$(47)^3$	70720	62100	.1219	1.18
	.051	$(37)^3$	70816	66340	.0632	1.25
	.00025	$(65)^3$	71860	Δ=12	.00017	2.36
Au C12 (5:1)						
	.512	$(80)^3$	30414	14784	.5139	1.00
	.405	$(74)^3$	30146	17768	.4106	1.01
	.301	$(62)^3$	30208	20636	.3174	1.05
	.205	$(59)^3$	30128	23240	.2286	1.12
	.104	$(47)^3$	30178	26528	.1209	1.16
	.051	$(37)^3$	30152	28324	.0806	1.20

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0.5V/47 kΩ series

$Au C_6$ (1:1)	.512	$(80)^3$	192256	104194	.4220	.824
	.405	$(74)^3$	182514	122364	.3296	.814
	.301	$(67)^3$	183244	136202	.2567	.853
	.205	$(59)^3$	184416	151786	.1769	.863
	.104	$(47)^3$	185143	167148	.0972	.935
	.051	$(37)^3$	185482	175898	.0522	1.03
$Au C_6$ (5:1)	.512	$(80)^3$	176334	94686	.4630	.904
	.405	$(74)^3$	177030	113180	.3607	.891
	.301	$(67)^3$	177306	127760	.2794	.928
	.205	$(59)^3$	177346	142018	.1992	.972
	.104	$(47)^3$	177946	158712	.1081	1.04
	.051	$(37)^3$	179400	167222	.0574	1.13
$Au C_6$ (3:1)	.512	$(80)^3$	87934	39274	.5534	1.08
	.405	$(74)^3$	88710	48574	.4524	1.12
	.301	$(67)^3$	89108	56886	.3616	1.20
	.205	$(59)^3$	89388	65548	.2667	1.30
	.104	$(47)^3$	89862	76284	.1510	1.45
	.051	$(37)^3$	89998	82512	.0822	1.62
	.000125	$(05)^3$	89832	Δ:130	.000033	2.64
$Au C_6$ (5:1)	.512	$(80)^3$	56102	24580	.5619	1.10
	.405	$(74)^3$	55896	30342	.4572	1.13
	.301	$(67)^3$	55844	35702	.3607	1.20
	.205	$(59)^3$	56176	41376	.2636	1.27
	.104	$(47)^3$	56056	47900	.1455	1.40
	.051	$(37)^3$	56890	51742	.0767	1.51
	.000125	$(05)^3$	56768	Δ:16	.000028	2.24

5.0V/10 MΩ series

$Au C_6$ (1:3)	.512	$(80)^3$	203100	115090	.4333	.846
	.405	$(74)^3$	209954	135510	.3546	.896
	.301	$(67)^3$	212360	152768	.2806	.932
	.205	$(59)^3$	216044	171782	.2049	1.00
	.104	$(47)^3$	218466	194116	.1115	1.07
	.051	$(37)^3$	219352	206616	.0581	1.15
$Au C_6$ (1:3)	.512	$(86)^3$	35382	29858	.1561	.305
	.405	$(74)^3$	35370	31130	.1199	.296
	.301	$(67)^3$	35252	32162	.0877	.291
	.205	$(59)^3$	35352	32200	.0609	.297
	.104	$(47)^3$	35488	34394	.0311	.299
	.051	$(37)^3$	35544	34976	.0160	.316
$Au C_6$ (1:1)	.512	$(80)^3$	45138	37050	.1792	.350
	.405	$(74)^3$	45386	38986	.1409	.348
	.301	$(67)^3$	45370	40658	.1039	.345
	.205	$(59)^3$	45394	42086	.0729	.356
	.104	$(47)^3$	45404	43682	.0377	.353
	.051	$(37)^3$	45344	44474	.0192	.379

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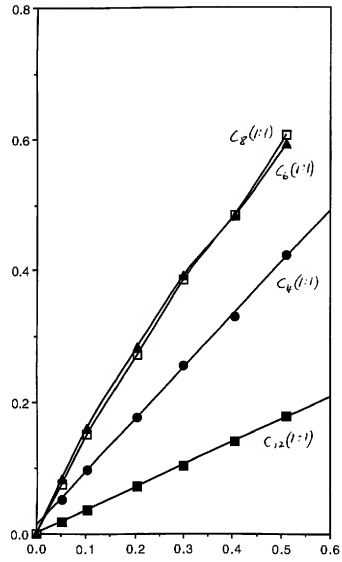
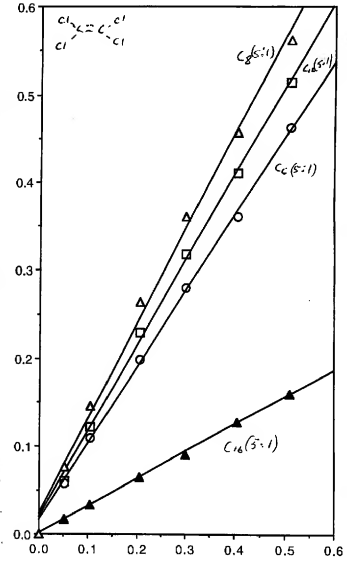
Witness _____ Date _____

Au C_{12} (3:1)	.512	$(80)^3$	133000	88766	.3702	.723
	.405	$(74)^3$	132836	92964	.3002	.741
	.301	$(67)^3$	133656	104124	.2212	.735
	.205	$(59)^3$	133358	112298	.1579	.770
	.104	$(47)^3$	134220	123324	.0808	.777
	.051	$(37)^3$	133744	128258	.0410	.809
Au C_{16} (5:1)	.512	$(80)^3$	52612	44190	.1601	.313
	.405	$(74)^3$	52702	45998	.1272	.314
	.301	$(67)^3$	53542	47816	.0899	.299
	.205	$(59)^3$	52652	49240	.0648	.316
	.104	$(47)^3$	52598	50846	.0333	.320
	.051	$(37)^3$	52748	51834	.0173	.342





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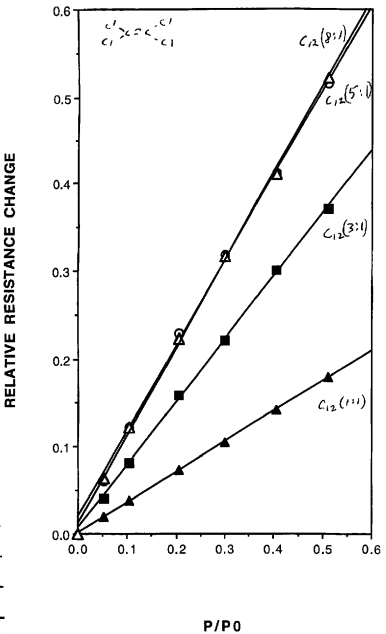

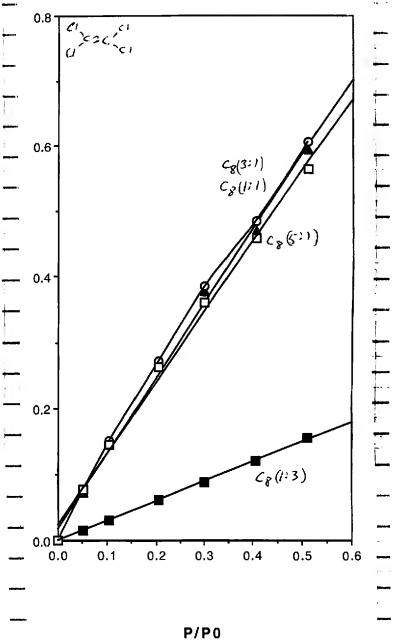

Response Table $\left(\frac{\Delta R/R}{(P/P_0)_{.20}}\right)$

C_n	1:3	1:1	3:1	5:1	8:1
C_{16}				.320	
C_{12}		.363	.777	1.16	1.18
C_8	.299	1.45	1.42	1.40	
C_6	1.07	1.55	1.45	1.09	
C_4		.935			

Read and understood (obtain two signatures):

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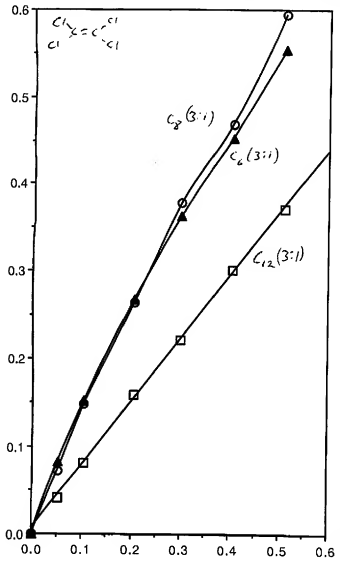

In slight contrast to toluene vapor, (NRL 7550-73) the C6 system is slightly more responsive to $CCl_2=CCl_2$ than the C8 system

Read and understood (obtain two signatures):

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An C_{12} (1:1) 20→120→20°C 5V DC stability

Objective: Determination of effect of a 5.0 V bias on a 32 pass An C_{12} (1:1) film conductivity through the 20°C (1hr) → 120°C (1hr) → 20°C (1hr) cycle in comparison with the 0.05 V DC (N7842-53) and 5.0 volt AC (N7850-89) experiments.

An MOS 302 device was plasma cleaned and coated with a An C_{12} 1:1 (10 mg/ml) air brushed deposition (32 passes) at 120°C and had an initial current of 0.36 nA (50 mV). The device was mounted on the substrate heater.

20°C

Time	I (nA)	Time	I (nA)	Time	I (nA)
18 45 0	32.30	19 46 61	161.83	20 46 121	182.5
18 46 1	25.67	19 47 62	168.00	20 47 122	140.02
18 47 2	35.26	19 48 63	169.88	20 48 123	114.62
18 48 3	35.96	19 49 64	171.98	20 49 124	99.34
12 49 4	34.96	19 50 65	173.47	20 50 125	89.58
12 50 5	34.89	19 51 66	175.26	20 51 126	82.88
18 55 10	34.78	19 52 67	177.11	20 52 127	78.51
19 00 15	34.84	19 53 68	178.94	20 53 128	75.56
19 05 20	34.86	19 54 69	180.89	20 54 129	73.34
19 10 25	34.84	19 55 70	182.61	20 55 130	71.83
19 15 30	34.90	20 00 75	192.12	20 00 135	68.18
19 20 35	34.91	20 05 80	200.10	21 05 140	67.00
19 25 40	34.90	2010 85	209.4	21 10 145	66.31
19 30 45	34.89	2015 90	218.1	21 15 150	66.08
1935 50	34.93	2020 95	226.4	21 20 155	65.79
1940 55	35.08	2025 100	235.2	21 25 160	65.57
1945 60	35.20	2030 105	242.7	21 30 165	65.35
20 → 120°C		2035 110	250.7	21 35 170	65.12
		2040 115	258.0	21 40 175	64.88
		2045 120	264.8	21 45 180	64.70
		120 → 20°C			

Read and understood (obtain two signatures):

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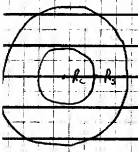
Calculations Related to Cluster Shell Volume and Thickness

Objective: Test relevancy of spherical model to Au C_n (X:Y) series.

Determine ligand shell thickness (R_s) from knowledge of core radius (R_c) and densities (ρ_c and ρ_s) and mass fractions (X_c and X_s) of core and ligand



$$X_s = \frac{M_s}{M_s + M_c} \qquad X_c = \frac{M_c}{M_s + M_c} \Rightarrow M_s = \frac{X_s}{X_c} M_c$$

$$V_s = \frac{M_s}{\rho_s} \qquad V_c = \frac{M_c}{\rho_c}$$

$$V_c = \tfrac{4}{3}\pi R_c^3 = \frac{M_c}{\rho_c} \Rightarrow M_c = \tfrac{4}{3}\pi R_c^3 \rho_c$$

$$V_s = \tfrac{4}{3}\pi (R_c + R_s)^3 - \tfrac{4}{3}\pi R_c^3 = \frac{M_s}{\rho_s} = \frac{1}{\rho_s}\frac{X_s}{X_c} M_c$$

$$\tfrac{4}{3}\pi (R_c + R_s)^3 - \tfrac{4}{3}\pi R_c^3 = \tfrac{4}{3}\pi R_c^3 \frac{\rho_c}{\rho_s}\frac{X_s}{X_c}$$

$$(R_c + R_s)^3 = \left(\frac{\rho_c}{\rho_s}\frac{X_s}{X_c} + 1\right) R_c^3$$

$$\left(\frac{R_c + R_s}{R_c}\right)^3 = \left(\frac{\rho_c}{\rho_s}\frac{X_s}{X_c} + 1\right)$$

$$\boxed{R_s = \left\{\left(\frac{\rho_c}{\rho_s}\frac{X_s}{X_c} + 1\right)^{1/3} - 1\right\} R_c}$$

For the Au and C_n thiols

C_4	$\rho_s = .842$	Liquid density of Thiols	Au $\rho_c = 19.3$
C_6	$\rho_s = .838$		
C_8	$\rho_s = .843$		
C_{12}	$\rho_s = .845$		
C_{16}	$\rho_s = .848$		

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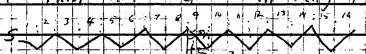
	Cn	1:3	1:1	3:1 (4:1)	5::1	8:1
	C_{16}		.6588		.8346	
	C_{12}	.7195	.7549	.7557 (.8427)	.8709	.8947
	C_8	.7507	.8065	.8411	.9041	
	C_6	.7922	.8241	.8835	.9192	
	C_4		.8787			
	C_{16}		.81		2.83	
	C_{12}	.86	1.14	1.56 (2.29)	2.97	3.61
	C_8	.63	1.11	1.53	3.01	
	C_6	.66	.96	1.61	2.92	
	C_4		1.24			
	C_{16}		1.09		2.18	
	C_{12}	.99	1.18	1.40 (1.69)	1.89	1.97
	C_8	.66	0.95		1.53	
	C_6	.60	0.78		1.30	
	C_4		0.78			

2.07
1.51
1.08
0.76
0.50

third extended chain length

Calculation of third extended chain length



$X = 1.54 \text{ Å} \sin \frac{109.4°}{2} = 1.26 \text{ Å}$

$C_{16} = 16 \times 1.26 = 2.07$ mm

$C_{12} = 12 \times 1.26 = 1.51$ mm

$C_8 = 8 \times 1.26 = 1.08$ mm

$C_6 = 6 \times 1.26 = 0.76$ mm

$C_4 = 4 \times 1.26 = 0.50$ mm

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(3) Bondi Volume Equivalents Calculation V_B

Tables 14.12 14.1 14.1

$-S-(CH_2)_n-CH_3$

10.8	(10.23)$_n$	13.67	$cm^3/mole$	$\times \left(\frac{10^7 nm}{cm}\right)^3 \left(\frac{mole}{6.02 \times 10^{23}}\right)$
.0179	(.0170)$_n$	.0227	$nm^3/molecule$	
		.0227		

C_n	16	12	8	6	4
V_B $nm^3/molecule$	.3126	.2746	.1766	.1426	.1086

(4) Number of thiol ligands bonded to surface of Cluster N_S

$$N_S = 4\pi R_c^2 \rho_{HCP} \gamma \qquad \rho_{HCP} = 13.89 \text{ atoms}/nm^2$$

$\gamma = 0.66$ thiol bound/surface gold atom

example: C8 1:1

$$N_S = 4\pi (1.11\,nm)^2 \; 13.89 \frac{atoms}{nm^2} \times \frac{.1568 \; RSH}{Au \; atom} = 139 \text{ molecules RSH}$$

(5) Van der Waals Volume V_{VDW} Calculation

$$V_{VDW} = N_S V_B$$

$$= 139 \times .1766 \; nm^3/molecule = 24.6 \; nm^3$$

(6) liquid density molecular volume of free thiols V_P

$$V_P = \frac{N_S}{\rho_{mol}}$$

$$= 139 / 0.893 \; g/cm^3 \left(\frac{cm}{10^7 nm}\right)^3 \times \frac{mole}{146.3 \; g} \times \frac{6.02 \times 10^{23} molec}{mole} = \frac{139 \; molec}{3.4 \; molec/nm^3}$$

$$= 40.1 \; nm^3$$

(7) Free volume fraction f

$$f = 1 - \frac{V_{VDW}}{V_P} = 1 - \frac{24.6}{40.7} = .387$$

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Arthur Ulman Date 5-19-97

Witness _____ Date _____

8) ~~Van Der Waal~~ Bondi density of C_n thiols ρ_B

$$\rho_B = \frac{MW}{V_R}\Big|_{C_8} = \frac{146.3\ g/mole}{176.6\ nm^3/molecule \times \frac{6.02\times10^{23}\ molec}{mole} \times \left(\frac{cm}{10^7\ nm}\right)^3}$$

	C_{16}	C_{12}	C_8	C_6	C_4
MW	258.5	202.4	146.3	118.24	90.19
ρ_B	1.37	1.37	1.38	1.38	1.38

9) Recalculation of R_{shell} using $\rho_s = 1.38$

$$R_s = \left[\left(\left(\frac{\rho_c}{\rho_s}\right)\left(\frac{x_s}{x_c}\right)+1\right)^{1/3} - 1\right] R_c$$

S

$\rho_s = 1.38$

C_n	1:3	1:1	3:1	(4:1)	5:1	8:1	extended chain
C_{16}		.83			1.58		2.02
C_{12}	.74	.88	1.03	(1.22)	1.35	1.38	1.51
C_8	.49	.70	.82		1.07		1.08
C_6	.44	.56	.67		.89		.76
C_4		.53					.50

Read and understood (obtain two signatures):

Witness______ Date______ Signature Arthur Snow Date 5-20-97

Witness______ Date______

Au S $C_2\phi$ (1:1)

$HAuCl_4$ +  $\xrightarrow[NaBH_4]{(C_8H_{17})_4NBr}$ $Au:C_2\phi$ (1:1)

Reagents

$(C_8H_{17})_4NBr$	3.42 g	(FW: 547)	6.25 mmol	Aldrich used as rec
Toluene	125 ml			Aldrich used as rec
$HAuCl_4 \cdot 3H_2O$	.5623 g	(FW: 354)	1.43 mmol	Aldrich used as rec
N_2O	47 ml			3x distilled
$C_6H_5CH_2CH_2SH$	.1982 g	(FW: 138.32)	1.43 mmol	Aldrich used as rec
Toluene	~0.5 ml			Aldrich used as rec
$NaBH_4$	.5912 g	(FW: 37.8)	15.6 mmol	Aldrich used as rec
N_2O	35.9 ml			3x distilled

Objective: Preparation aromatic alkyl thiol stabilized gold cluster to determine IR aromatic functionality & Vapor response characteristics in microsensor study.

The solutions were prepared as indicated above. The $HAuCl_4/N_2O$ was prepared and handled in acid washed glassware.

To the $(C_8H_{17})_4NBr$/Toluene solution in a 500 ml Erlenmeyer was added the $HAuCl_4/N_2O$ solution with rapid stirring. After 1 min color disappearance from aqueous phase indicated transfer of the $HAuCl_4$. The ϕC_2SH was added with stirring - no significant color change occurred. With rapid stirring the $NaBH_4/N_2O$ was added (~1 sec) the mixture turned purple-black with gas effervescence. Rapid stirring was continued for (9:45 → 12:45) 3 hrs.

The reaction was worked up by transfer to a 500 ml sep funnel and separation of the toluene phase in a 250 ml round bottom. This was concentrated (55°c/60 mm) to a 5-10 ml volume and ppt

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Arthur Ahn Date 5-21-97

Witness _____ Date _____

into 600 ml rapidly stirred MeOH. After settling for 1 hr, a clean separation occurred. The supernate was decanted and crude product collected by centrifugation. After drying at 30°C the product was redissolved in 5 ml toluene and added dropwise into 200 ml rapidly stirred MeOH

After standing overnight at 10°C the supernate was quite dark but centrifuging rendered a nearly colorless supernate. This is one of the cleanest separations on the 2nd NPT to date.

The product was collected by centrifugation, dried at 30°C and vacuum dried to yield 0.3146 g

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 5-22-97

Witness _____ Date _____

Au $C_2\phi$ 1:1 Toluene Isotherm

Objective: Obtain Toluene Vapor Isotherm for comparison with Aliphatic Au Cn (1:1) series

A 10 mg/ml stock solution (2 Wells) was prepared and air brushed onto a freshly plasma cleaned MSI 302 device (16 passes) 5th - 50 mV current was 1.6 μA. Inspection with the optical microscope showed good coverage but ~ slightly more coarse texture

Initial Toluene vapor exposures at P/P_0 .512 (.80)^3 on Au $C_2\phi$ (1:1) and Au C_8 (1:1) indicated the Au $C_2\phi$ had a slower response and 30, 60 and 120 exposure time cycles were run

exposure time	P/P_0	dilution factor	Baseline	signal	signal/baseline	$\Delta R/R_0$
Au C_8 (1:1) 5V/100 kΩ						
30 sec	.512	80 80 80	20754	17330	.16502	
60 sec	.512	80 80 80	20894	17380	.16468	
120 sec	.512	80 80 80	20784	17380	.16449	
120 sec	.405	74 74 74	20688	9106	.15598	
Au $C_2\phi$ (1:1) 0.5V/100 kΩ						
30 sec	.512	80 80 80	42452	24952	.41212	
60 sec	.512	80 80 80	41744	22428	.46115	
120 sec	.512	80 80 80	42064	21542	.48879	.593
120 sec	.405	74 74 74	42164	24288	.42408	1.047
120 sec	.301	67 67 67	41796	26844	.35777	1.188
120 sec	.205	59 59 59	41740	26940	.28227	1.379
120 sec	.1064	47 47 47	41904	33862	.19179	1.845
120 sec	.05567	37 37 37	42058	36806	.12349	2.463
120 sec	.0244	29 29 29	42112	38857	.07741	3.173
120 sec	.01338	24 24 24	42170	39938	.05293	3.835
120 sec	.00803	20 20 20	42262	40704	.03687	4.608
120 sec	.00460	16 16 16	42314	41332	.02321	5.660

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Witness _____ Date _____ Signature Arthur Chu Date 5-23-97

Witness _____ Date _____

120 sec	.00173	12	12	12	42352	41830	.01233	7.124
120 sec	.000512	8	8	8	42360	42172	.004438	8-668 } repeat
120 sec	.000125	5	5	5	42366	42312	.001275	10.20

$CCl_2 = CCl_2$ Is. thkn

Au $C_2\phi$ (1:1) 0.5V/100 KΩ

120 sec	.512	80 80 80	41438	21860	.4725	.923	
120 sec	.405	74 74 74	41438	24486	.4091	1.010	
120 sec	.301	67 67 67	41572	27686	.33140	1.110	
120 sec	.205	59 55 55	41856	30.594	.2691	1.313	
120 sec	.104	47 47 47	41958	35.044	.1648	1.584	
120 sec	.0587	37 37 37	42208	37754	.10555	1.798	
120 sec	.0244	25 29 29	42406	39662	.06471	2.652	
120 sec	.0138	24 24 24	42528	40722	.04267	3.077	
120 sec	.001805	20 20 20	42644	41438	.02838	3.535	
120 sec	.000416	16 16 16	42592	41876	.01681	4.1100	
120 sec	.000173	12 12 12	42606	42264	.008027	4.6401	
120 sec	.000512	8 8 8	42620	42548	.002648	5.4133	
120 sec	.000125	5 5 5	42628	42580	.000891	7.13	

$CH_3CH_2CH_2OH$ Is. thkn (Au: $C_2\phi$ (1:1))

720 sec	.512	80 80 80	43082	41576	.03496	.06827
120 sec	.405	74 74 74	43052	42002	.02439	.060212
120 sec	.301	67 67 67	42968	42274	.01615	.05366
120 sec	.205	59 55 55	42920	42488	.009832	.04796
120 sec	.104	47 47 47	42836	42634	.004716	.04534
120 sec	.0587	37 37 37	42774	42660	.002665	.05247
120 sec	.0244	25 29 29	42720	42650	.001639	.06715
120 sec	.0138	24 24 24	42668	42624	.001031	.07473

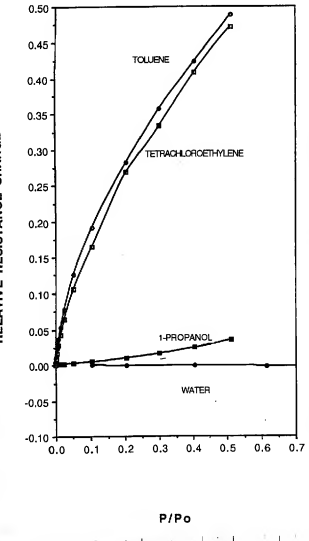
N_2O Is. thkn (Au $C_2\phi$ (1:1))

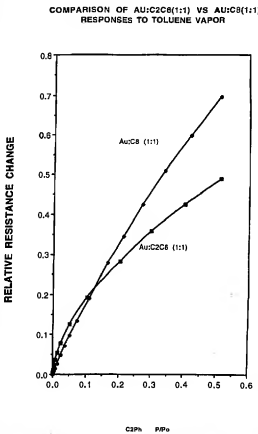
120 sec	.804	93 93 93	42478	-4.5x25	-.00265	
120 sec	.674	85 85 85	42578	~~-10~~ -4.9x25	-.00196	
120 sec	.405	74 74 74	42430	-4.9x20	-.00090	
120 sec	.205	59 59 59	42388	-4.9x20	-.00047	
120 sec	.104	47 47 47	42318	0	0	

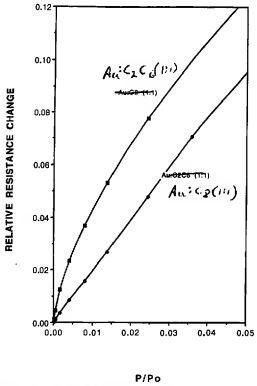
Read and understood (obtain two signatures):

Witness Date Signature Date 5-27-97

Witness Date







ad and understood (obtain two signatures):

itness Date Signature Date 6-15-99

itness Date

Au 5 C, ϕ (1:1)

$HAuCl_4$ + ⓒ CN_2SH $\xrightarrow[NaBH_4]{(C_8H_{17})_4NBr}$ Au: C, ϕ (1:1)

Reagents

$(C_8H_{17})_4NBr$	3.425	(FW: 547 g/mol)	6.25 mmol	Aldrich (used as rec)
Toluene	125 ml			Aldrich (used as rec)
$HAuCl_4 \cdot 3H_2O$	0.5632 g	(FW: 394 g/mol)	1.43 mmol	Aldrich (used as rec)
Water	47 ml			3x distilled
$C_6H_5CH_2SH$	0.1772	(FW: 124.21 g/mol)	1.43 mmol	Aldrich used as rec
Toluene	~ 0.5 ml			Aldrich used as rec
$NaBH_4$	0.5906	(FW: 37.83 g/mol)	15.6 mmol	Aldrich used as rec
H_2O	35.5 ml			3x distilled

Objective: Preparation of benzyl thiol stabilized gold cluster for comparative Vapor response measurement with Au: $C_2\phi$ and Au: C_8 (1:1) systems in microsensor study

Solutions were prepared as indicated above. The $NAuCl_4/H_2O$ soln was protected and handled in acid washed glassware.

To the $(C_8H_{17})_4NBr$/Tol soln in a 500 ml Erlynmeyer was added the $HAuCl_4/H_2O$ with rapid stirring. After 2 min when color of toluene and H_2O phases indicated phase transfer had occurred the ϕCH_2SH/toluene was added (no significant color change observed). With more rapid stirring the $NaBH_4/H_2O$ was added (CAUTION) with observation of purple-black coloration and effervescence. Rapid stirring was continued for (13:20 → 16:20) 3 hr.

The reaction was worked up by transferring to a 500 ml sep funnel, and separating the toluene phase into a 250 ml round bottom

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 5-27-97

Witness _____ Date _____

flask. This was concentrated to a ~10-12 ml volume and ppt by dropwise addition into 600 ml rapidly stirred MeOH.

After 3 hr stand at rm temp a good settling occurred. The supernate was decanted and crude product collected by centrifugation. This product was dissolved in ~4 ml (not quite soluble in 3 ml) and ppt into 200 ml rapidly stirred MeOH. Rapid settling did not occur in this and suspension was allowed to stand at 10°C overnight.

Supernate remained highly colored. Centrifugation at 6000 rpm/2 min was attempted - supernate remained very dark. At 6000 rpm/10 min produced a better result with a transparent but dark supernate. The product was collected by 6000 rpm/10 min centrifugation. The product was dried at 30°C in vacuum desiccator for 24 hr. yield 0.265 g

ead and understood (obtain two signatures):

itness _____ Date _____ Signature Arthur Ohm Date 5-27-57

itness _____ Date _____

Au:C_{12} (4:1) 3rd Reppt Purification and Temperature-Conductivity Measurements

Objective: Obtain better Temp-Conductivity data on Au C_{12}(4:1) - The unstability of the measurements (N7250-75 & N7250-42) appeared to indicate that the sample (N7250-40) may lack sufficient purity - possibly some ~~(C6H13)4NBr~~ contamination and a third reprecipitation into MeOH might improve stability of these measurements. The entire product (N7250-40) was dissolved in ~3 ml/ toluene & ~~and~~ added dropwise into ~200 ml rapidly stirred, freshly opened MeOH. After standing overnight at 10°C, the supernate remained highly colored. Centrifuging for 15 min/6000 rpm produced a low quality separation with the product poorly packed at the bottom of the tube and a strongly colored but transparent supernate. It was decided to attempt product collection with these conditions. During the last centrifuge spin the tube failed but product could be recovered. This was transferred to the original vial and vacuum dried.

A sample was prepared for -76 → 40°C conductivity measurements. A 10.0 mg/ml Au:C_{12}(4:1)/CHCl$_3$ solution was prepared and air brushed ~~16 passes~~ onto ~120°C MSI 302 (plasma cleaned) device.

~~Heat Cycle~~ Initial current 50 mV ÷ 17 nA.

I(nA)	T(°C)	I(nA)	T(°C)	I(nA)	T(°C)	I(nA)	T(°C)	I(nA)	T(°C)	
4.775	-76.2	7.450	-57.9	10.819	-38.0	15.052	-10.1	16.209	14.8	
5.030	-74.2	7.710	-56.5	11.159	-35.9	15.135	-9.1	17.030	17.4	
5.285	-72.6	7.892	-55.3	11.375	-35.0	15.223	-8.1	17.381	22.3	slow
5.527	-71.0	P.143	-53.8	11.552	-34.0	15.413	-5.8	17.67	27.4	drift
5.643	-70.2	8.387	-52.2	11.735	-33.0	15.472	-5.0	17.93	29.3	
5.783	-69.0	8.610	-50.8	11.919	-32.0	15.724	-3.0	18.64	34.7	
6.101	-67.0	8.894	-49.0	12.175	-30.6	15.928	-0.1	18.24	38.1	
6.261	-66.0	9.135	-47.5	12.884	-25.7	16.010	2.6	18.45	44.1	
6.426	-65.0	9.483	-45.9	13.264	-23.9	16.151	5.1			
6.636	-63.5	9.657	-44.5	13.476	-22.6	16.231	7.2			
6.869	-62.0	9.859	-43.3	13.733	-20.9	16.367	9.0			
7.095	-60.5	10.148	-41.6	14.045	-18.6	16.505	11.2			
7.321	-59.0	10.654	-39.0	14.944	-11.2	16.655	13.1			

slow heating

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 5-29-97

Witness _____ Date _____

20° → 120 → 20°C Thermal Cycle Stability expt

Same sample as Variable temperature expt (Preceding Page)

time	I (mA)	time	I (mA)	time	I (mA)
20:30	17.40	21:31	20.83	22:31	29.03
20:31	17.39	21:32	20.86	22:32	28.57
20:32	17.38	21:33	19.95	22:33	27.60
20:33	17.41	21:34	20.72	22:34	26.66
20:34	17.42	21:35	20.46	22:35	25.91
20:35	17.43	21:36	20.70	22:36	25.35
20:40	17.44	21:37	20.93	22:38	24.75
20:45	17.50	21:38	21.18	22:40	24.48
20:50	17.60	21:39	21.41	22:45	24.31
20:55	17.70	21:40	21.62	22:50	24.26
21:00	17.68	21:45	22.54	22:55	24.32
21:05	17.74	21:50	23.24	23:00	24.35
21:10	17.85	21:55	24.01	23:05	24.47
21:15	17.93	22:00	24.73	23:10	24.58
21:20	17.97	22:05	25.43	23:15	24.69
21:25	18.03	22:10	26.01	23:20	24.84
21:30	18.12	22:15	26.52	23:25	24.86
20°C → 12.0°C		22:20	26.88	23:30	25.00
		22:25	27.29		
		22:30	27.62		
		12.0°C → 20°C			

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature [signature] Date 5-23-97

Witness _____ Date _____

$Au\ SC_1\phi OCH_3$ (1')

$HAuCl_4 + CH_3O\text{-}\phi\text{-}CH_2SH \xrightarrow[NaBH_4]{(C_8H_{17})_4NBr} Au:C_1\phi OCH_3$

Reagents

$(C_8H_{17})_4NBr$	3.42 g	(FW: 547 g/mol)	6.25 mmol	Aldrich used as rec'd
Toluene	125 ml			Aldrich used as rec'd
$HAuCl_4 \cdot 3H_2O$	.559	(FW: 394 g/mol)	1.42 mmol	Aldrich used as rec'd
Water	47 ml			3 × distilled
$pCH_3O\ C_6H_4CH_2SH$	.218 g	(FW: 154.23 g/mol)	1.42 mmol	Aldrich used as rec'd
Toluene	~1 ml			Aldrich used as rec'd
$NaBH_4$	.5863	(FW: 37.8 g/mol)		Aldrich used as rec'd
Water	39 ml			3 × distilled

Objective: Preparation of the methoxy functionalized benzyl thiol gold cluster for comparative vapor response measurement with the more hydrophobic cluster system.

Solutions were prepared as indicated above. The $HAuCl_4/H_2O$ solution was prepared and handled in acid washed glassware.

To the $(C_8H_{17})_4NBr$/Toluene solution in a 500 ml Erlenmeyer was added the $HAuCl_4/H_2O$ solution with rapid stirring. After ~2 min. when color of aqueous (clear) and toluene (dark brown) phases indicated Au(I) transfer had occurred. The $CH_3O\phi CH_2SH$ was added - no observable color change observed. With vigorous rapid stirring the $NaBH_4/H_2O$ solution was added over a 15 sec interval. Within a purple-black coloration developed and milky opalescence of hydrosol. Rapid stirring was continued for (13:30 → 16:10) 3 hr.

The solution was worked up by transferring to a 500 ml

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Arthur Chow Date 6-2-97

Witness _____ Date _____

Sep Funnel and separating the toluene phase into a 250 ml round bottom flask. This was concentrated to a ~10 ml volume (55°C/60 Torr) and ppt by dropwise addition into 600 ml MeOH.

After 4hr standing a good settling had occurred and a relatively clear supernate was decanted and the crude product collected by centrifugation and dried at 30°C. This crude product was redissolved in 3.5 ml toluene and reppt by dropwise addition to 200 ml rapidly stirred MeOH. A clouded suspension formed and was placed in a refrigerator overnight (10°C)

After standing 10 hr at 10°C the supernate remained highly colored. Centrifugation of the supernate (5000 rpm/30 min) separated much of the solid leaving a brown but transparent supernate. The purified product was isolated by centrifuging the 200 ml, and finally washed with ~5 ml MeOH, centrifuged, dried at 35-40°C transferred to a vial and vacuum dried overnight yield 0.2830 g

It was observed during cleanup that this cluster complex is significantly soluble in acetone.

ad and understood (obtain two signatures):

tness Date Signature Date 6-2-97

tness Date

Detection of Trace Organic in Aqueous Condensed Phase with Au:C8 1:1 Chemiresistor

Objective: Demonstration that this alkanethiol stabilized gold cluster chemiresistor will respond to trace organic dissolved in water. The alkanethiol stabilized gold clusters are very hydrophobic, and hypothesis is that there will be a strong chemical potential for a dissolved trace organic such as toluene to partition into the cluster deposition which should respond in similar manner as to toluene vapor. The following experiment is designed to demonstrate this phenomenon and possibly the sensitivity.

The Au:C8(1:1) cluster was selected as a very toluene sensitive candidate with strong hydrophobicity.

Au:C8 1:1 / MSI 302 devices were prepared as follows. The (4) MSI 302 devices were inspected (50x), blanked for shorts and plasma cleaned.

A 10 mg/ml stock solution of Au:C8(1:1) N7542-41 was prepared. Two of the devices were mounted on a resistive heater, heated to 120°C and sprayed with an airbrush (finest nozzle setting) with 16 1-sec pulses. The second pair of devices were done in likewise fashion. Current measurements (50mV) were made in each device and are designated as follows:

Au:C8 1:1 #1	Au:C8 1:1 #2	Au:C8 1:1 #3	Au:C8 1:1 #4
33.7 nA	28.1 nA	24.6 nA	33.7 nA

Read and understood (obtain two signatures):

Witness Date Signature Date 6-2-97

Witness Date

A saturated solution of toluene in water was prepared by placing ~4.5 ml water (3x dist. H$_2$O) in a 2.5 ml vial and adding ~4 drops Toluene (99.9+% Aldrich) and stirring gently for ≥3 hr. The solubility of toluene in water is 0.0515 %W (Riddick & Bunger) A series of dilutions from this saturated solution were made as follows:

T_0 saturated 0.0515

T_1 $\frac{.2079 g_{T_0} (.0515)}{.2079 g_{T_0} + 18.76 g_{H_2O}} = 0.00531$

T_2 $\frac{.2219 g_{T_1} (.00531)}{.2219 g_{T_1} + 18.76 g_{H_2O}} = 0.000563$

T_3 $\frac{.2160 g_{T_2} (.000563)}{.2160 g_{T_2} + 18.68 g_{H_2O}} = 0.0000530$

The experimental test was conducted by placing a quantity of water or the T_0/H_2O solutions in a 10x75 mm tube up to a level to just immerse the electrode area

Au C_8 111 #9 33.1

H_2O immersion 23.14 nA in AIR @ 50mV DC

~~51.5 nA in Dist. H_2O~~

~~35.42~~ ~~34.81~~ nA in AIR

T_3 ~45 nA ±3 in .0000530% Toluene

(120nA - 140nA) in Dist. H_2O (drifting down)

~32 nA in AIR

T_2 137 nA ± in T_2 .000563 %

28.7 nA in AIR

133 ± 2 nA in H_2O

28.7 nA in AIR

T_1 110 nA in T_1 .00531% (drifting down)

30.2 nA in AIR

125 nA in Dist. H_2O

26 nA in AIR

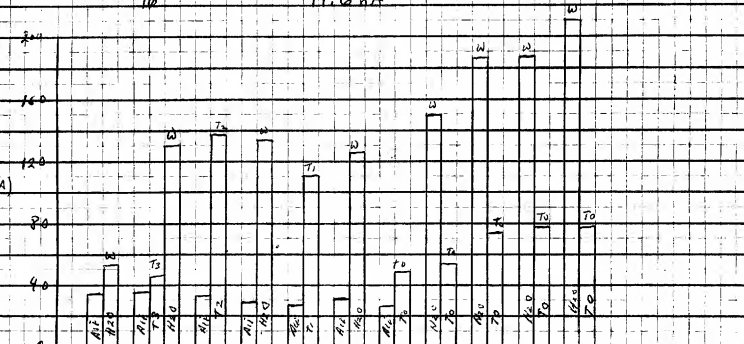
T_0 48 nA in T_0 0.0515 %

Read and understood (obtain two signatures): TØ

Witness Date Signature Date 6-2-77

Witness Date

DIST H_2O – 149 nA
TØ – 53 nA
DIST H_2O – 185 nA
TØ – 73.6 nA
DIST H_2O – 186 nA
TØ – 78.4 nA
DIST H_2O – 210 nA
TØ – 77.6 nA



It appears that water immersion after a moderate conditioning time results in an increase in conductivity of almost one of magnitude (i.e. 30 nA → 200 nA). Removal from the water and a min of two to dry reverts the conductivity to the 25-30 nA range. Immersion in a toluene saturated water solution (ca. 0.05% w/w) causes the conductivity to decrease to the 70-80 nA level on at least 3 to four repetitions. Clearly the following causes a strong decrease in conductivity. This cycling causes some deterioration of the film. Optical microscopy observations show a lot of microcracking in addition to lift of several portions of the film. Treatment of the film with a diethiol (eg $HS(CH_2)_2SH$) might save this problem

Read and understood (obtain two signatures):

Witness Date Signature Arthur Hunt Date 6-3-97

Witness Date

I-V Curve data on $Au\,C_n(X:4)$

Objective - Collective summary of ±2 V I-V Ohms law demonstration for devices prepared for utilization in Vapor response study ie N7550-38 etc

V(volts)	$C_{12}(1:3)$	$C_{12}(1:1)$	$C_{12}(3:1)$	$C_n(4:1)$	$C_n(5:1)$	$C_{12}(8:1)$	$C_{12}(6:1)$	$C_{12}(1:3)$	$C_{12}(1:1)$
			nA	nA	nA	nA	nA	nA	nA
0	-25 pA	-10 nA	.163	.589	3.55	9.48	.041	.014	3.98
.05	74 pA	.136 nA	1.48	5.33	31.59	83.92	.41	.160	35.2
.10	165 pA	.275	3.01	10.82	64.28	170.9	.83	.327	71.5
.20	.332 nA	1.35	6.06	21.95	129.76	343.7	1.67	.662	144.1
.30	.461 nA	2.17	9.22	34.40	198.25	526.2	2.55	1.007	218.7
.40	.601 nA	2.84	12.3)	49.28	262.5	700.7	3.39	1.335	291.1
.50	.728 nA	3.65	15.38	54.95	327.2	873.5	4.18	1.656	362.8
.60	.852 nA	4.30	18.43	65.6	392.7	10.48	5.01	1.980	435.2
.70	1.00 nA	4.98	21.68	77.2	461.4	12.31	5.88	2.321	511.1
.80	1.15 nA	5.60	24.81	88.9	527.0	1406	6.93	2.645	583.7
.90	1.28 nA	6.24	27.85	100.3	592.0	1579	7.54	2.967	655.2
1.00	1.40 nA	6.98	31.01	112.8	657.6	1754	8.36	3.300	727.4
1.2	1.78 nA	8.32	37.98	140.6	792.1	2114	10.11	4.001	875.1
1.4	2.11 nA	9.59	44.15	166.4	923.0	2462	11.82	4.707	1018
1.6	2.5) nA	11.07	50.80	194.6	1058	2871	13.61	5.459	1165
1.8	2.91 nA	12.54	57.35	22210	1192	3176	15.42	6.227	1309
2.0	3.32 nA	13.53	63.97	249.4	1327	3535	17.27	7.003	1454
0	-.28 pA	-.3 pA	.148	.51	0	9.48	.037	.010	3.88
-.05	-.103 pA	-.40	-1.40	-5.52	3.52	-78.06	-.40	-.151	-31.8
-.10	-.176 pA	-.78	-2.92	-11.35	-29.28	-165.5	-.82	-.307	-67.4
-.20	-.335 nA	-1.57	-6.12	-23.20	-62.06	-350.3	-1.70	-.634	-142.0
-.30	-.472 nA	-2.26	-9.17	-34.25	-130.7	-525.1	-2.52	-.943	-214.6
-.40	-.606 nA	-2.93	-12.19	-45.24	-156.2	-698.1	-3.35	-1.248	-285.1
-.50	-.733 nA	-3.62	-15.22	-56.41	-262.5	-873.0	-4.17	-1.556	-356.6
-.60	-.870 nA	-4.34	-17.45	-67.78	-329.0	-1056	-5.04	-1.832	-431.5
-.70	-.991 nA	-4.99	-21.50	-78.39	-396.6	-1231	-5.87	-2.196	-502.1
-.80	-1.07 nA	-5.60	-24.58	-89.35	-462.1	-1404	-6.69	-2.512	-574.3
-.90	-1.18 nA	-6.22	-27.67	-100.94	-526.9	-1580	-7.50	-2.835	-646.1
-1.00	-1.31 nA	-6.81	-34.99	-113.6	-592.5	-1763	-8.38	-3.178	-721.3
-1.2	-1.58 nA	-8.20	-37.48	-138.9	-661.4	-2112	-10.08	-3.839	-863.8
-1.4	-1.87 nA	-9.58	-44.08	-166.2	-792.0	-2471	-11.84	-4.538	-1010
-1.6	-2.17 nA	-10.90	-51.61	-193.6	-926.7	-2826	-13.60	-5.245	-1154
-1.8	-2.53 nA	-12.32	-57.26	-222.9	-10.60	-3185	-15.40	-5.9 80	-1300
-2.0	-2.89 nA	-13.90	-63.54	-250.1	-11.95	-3534	-17.22	-6.711	-1440
					-1327				
	m=1.46	m=672	m=31.6	m=120	m=661	m=1764	m=8.50	m=3.33	m=724
	b=.048	b=.026	b=.048	b=-.028	b=-.163	b=.554	b=.0088	b=.061	b=3.63
	V/I_{sc}=.033	I/I_{sc}=.004	V_0=-.0015	V_0=.00065	V_0=.00025	V_0=-.00051	V_0=.00010	V_0=.018	V_0=.0050
Sm	1.2×10^{-9}	5.6×10^{-9}	3×10^{-8}	1×10^{-7}	5×10^{-7}	1×10^{-6}	7×10^{-9}	3×10^{-9}	6×10^{-7}
.2M	2.3×10^{-9}	1.1×10^{-8}	5.0×10^{-8}	1.9×10^{-7}	1.1×10^{-6}	2.8×10^{-6}	1.4×10^{-8}	5.3×10^{-9}	1.2×10^{-6}

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 6-4-97

Witness _____ Date _____

Calculation of σ, Vapor response devices are 16 pass castings $\frac{1}{2}$($\frac{1}{2}$ 32 pass ×.4μ) .2μ

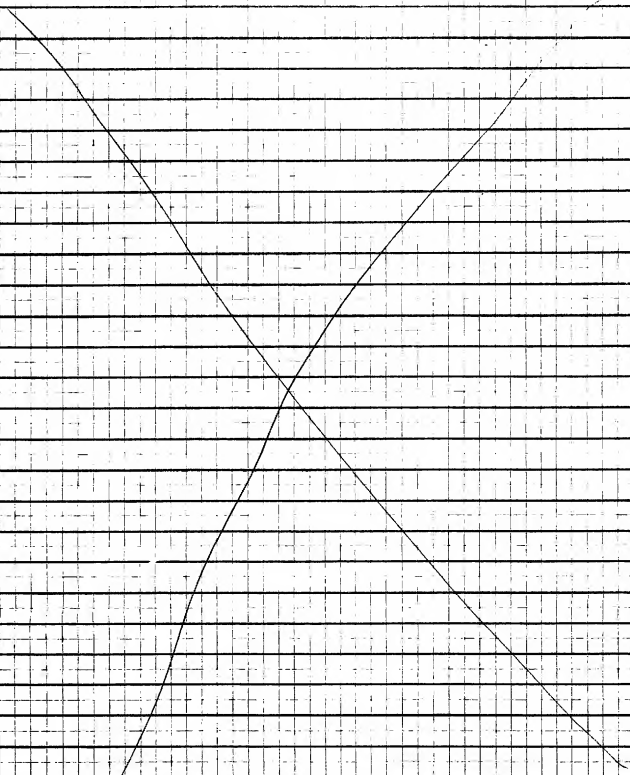
$$\sigma = \frac{d}{2(n-1)l}\,\frac{1}{h}\,\frac{I}{V} = \frac{15\mu}{2(50-1)\,4800\mu}\,\frac{1}{.2\mu\,\frac{cm}{10^4\mu}}\,\frac{I}{V} = 1.59\left(\frac{I}{V}\right)$$

(3:1)	C_8(5:1)	C_6(1:3)	C_6(1:1)	C_6(3:1)		C_6(5:1)	C_4(‡:1)	
nA	nA	nA	nA	nA		nA	nA 1188	
24.0	512	.274	18.73	782		2190	~~10629~~	
09.6	4530	2.44	166.0	6954		19446	10635	
27.3	9217	4.95	336.1	14137		39540	21650	
62.7	18593	9.96	677.2	28510		79740	43690	
318.6	28410	15.19	1034.5	43576		121830	66830	
754.3	37790	20.15	1376.2	57946		161990	88980	unstable
2187	47070	25.04	1715.7	72220		200600	110890	rapid increase
2623	56450	29.95	2060	86670		240700	133050	measurement
3083	66280	35.09	2423	101880		282800	156360	taken after 15 sec
3524	75680	40.00	2770	116450		322900	178700	
3960	84970	44.80	3113	130700		362500	199800	
403	94360	49.66	3462	144900	past stability	402600	223700	
5305	113600	59.65	4174	174900		484860	273100	
185	132290	69.45	4869	243000		564860	323000	
089	151580	79.52	5590	238300		647100	375500	
979	170640	89.45	6308	272500		728400	428100	
879	189940	99.50	7030	305900		810700	482200	
3.5	514	.255	18.02	817		2180	1282	
192.9	-4186	-2.13	-150.0	-6714		-17900	-10580	
09.6	-8886	-4.50	-319.5	-14219		-37530	-22420	
60.7	-18729	-9.47	-670.9	-29920		-79930	-47220	
291.2	-28110	-14.20	-1006.2	-44880		-119980	-77890	
717.5	-37400	-18.88	-1338.0	-59660		-159600	-94300	
149	-46800	-23.60	-1675.8	-74580		-199680	-117940	
602	-56640	-28.54	-2031	-90210		-243000	-142650	
036	-66030	-33.25	-2373	-105100		-283000	-166280	
467	-75330	-37.92	-2712	-119790		-322600	-189480	
904	-84730	-42.62	-3059	-134770		-362700	-214600	
365	-94580	-47.58	-3422	-150310		-404700	-240300	
245	-113280	-57.02	-4106	-179800		-484500	-286700	
143	-132550	-66.77	-4810	-211900		-566000	-334700	
030	-151590	-76.43	-5515	-243800		-647900	-384600	
928	-170890	-86.22	-6232	-276600		-730300	-437600	
803	-189640	-95.76	-6926	-308900		-810200	-488800	
400	m = 94700	m = 48.7	m = 3460	m = 150480		m = 404380	m = 237170	
204	b = 85.9	b = .895	b = 23.4	b = -1500		b = 144	b = -4071	
046	V_0 = -.00095	V_0 = -.018	V_0 = -.0068	V_0 = +.010		V_0 = -.00036	V_0 = +.017	
10^{-6}	8×10^{-5}	4×10^{-8}	3×10^{-6}	1×10^{-4}		3×10^{-4}	2×10^{-4}	
10^{-6}	1.5×10^{-5}	7.8×10^{-8}	5.5×10^{-6}	2.4×10^{-4}		6.4×10^{-4}	3.8×10^{-4}	

Read and understood (obtain two signatures):

Witness______ Date______ Signature Arthur Snow Date 6-4-97

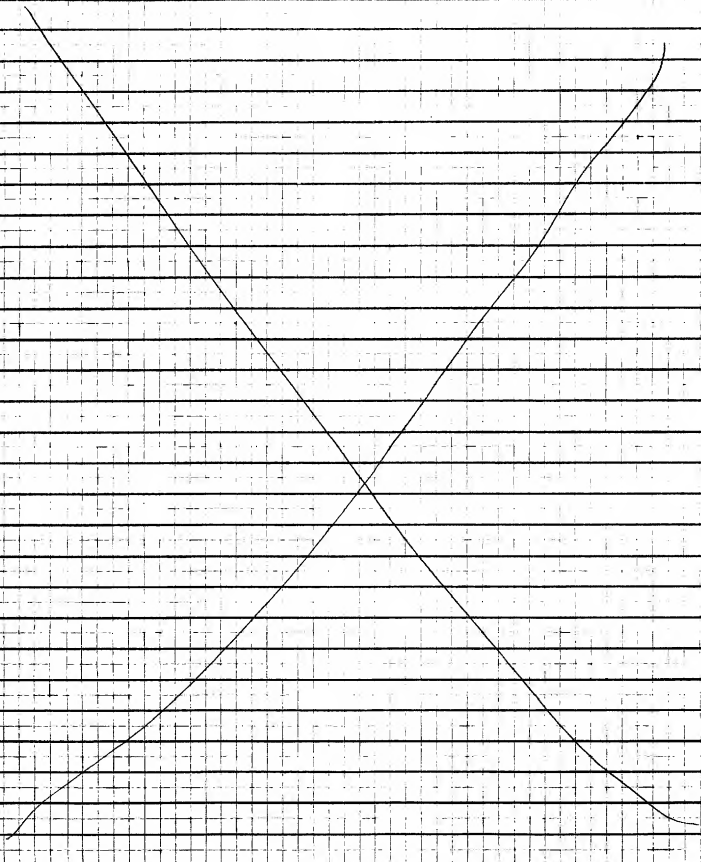
Witness______ Date______



Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date _____

Witness _____ Date _____



Read and understood (obtain two signatures):

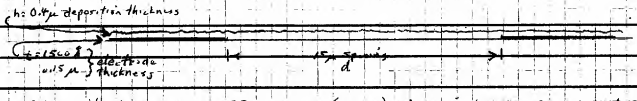
Witness _____ Date _____ Signature _____ Date _____

Witness _____ Date _____

Interdigital Electrode Analysis

Objective: Assessment of deposition thickness effect relative to electrode thickness and spacing.

Scale Perspective on MSI 302 device



Film thickness - 32 passes (1 sec) with air brush using 10 mg/ml / 50 / 50 in

Weight increase on device is 0.0009 g

device dimensions 12.5 mm × 7.0 mm × $\left(\frac{cm}{10mm}\right)^2$ = 0.875 cm^2

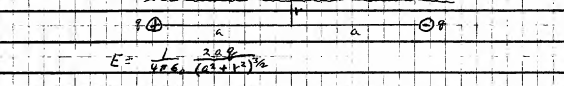
Thickness = $\frac{0.0009\,g}{0.875\,cm^2} \times \frac{cm^3}{3.06\,g} \times \frac{10^4\,\mu}{cm}$ = 0.4 μ

ρ 3.06 JACS 117, 12537 (1995) Au C_{12}(1:1)

$\sigma = \left(\frac{d}{2(n-1)\delta}\right)\frac{1}{h}\frac{I}{V} = \frac{15}{2(50-1)\,4\mu}\times\frac{1}{.4\mu\times\frac{cm}{10^4\mu}}\times\frac{1}{10.5\,V}$ = 15.94 (Tcomp)

Consider edge of electrode a point charge separated by a 15μ spacing

Then use Halliday & Resnick (p. 762) solution for an electric field surrounding a dipole



$E = \frac{1}{4\pi\epsilon_0}\frac{2aq}{(a^2+h^2)^{3/2}}$

at r=0 (in plane of electrode)

$E_1 = E_0 = \frac{1}{4\pi\epsilon_0}\frac{2q}{a^2}$

Then the ratio of the field E' at r=h to the field E_0 at r=0 is

$\frac{E'}{E_0} = \frac{\frac{1}{(a^2+h^2)^{3/2}}}{\frac{1}{a^3}} = \frac{a^3}{(a^2+h^2)^{3/2}} = \frac{1}{\left(\frac{a^2+h^2}{a^2}\right)^{3/2}} = \frac{1}{\left(1+\left(\frac{h}{a}\right)^2\right)^{3/2}}$

in our model $a = \frac{d}{2}$

$\frac{E'}{E_0} = \frac{1}{\left(1+\left(\frac{2h}{d}\right)^2\right)^{3/2}}$

Calculation for

h(μ)	0.4	1.0	2.5	5.0	7.5	10.0	15.0
d = 15μ and h varying from 0.4μ to 15μ E'/E_0	0.996	0.979	0.854	0.576	0.354	0.216	0.089
h/d	0.027	0.067	0.167	0.333	0.500	0.667	1.000

* Note as d becomes smaller, a thinner film is necessary to preserve the same level of accuracy e.g. d = 5μ | E'/E_0 = 0.963 at h = 0.4μ

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Anthony Sant Date 6-4-97

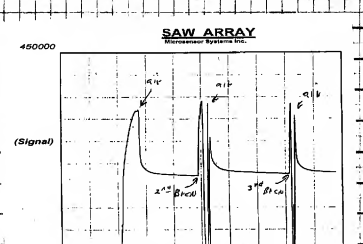
Witness _____ Date _____

Re: Cyanogen Bromide Response of Au-C8(11) Sensor

Objective: Determination if and to what degree the alkanethiol stabilized gold cluster chemiresistor sensor will respond to cyanogen bromide. The BrCN is a substitute for the chloride analog for which similar activity would be predicted. The long term objective is an effective sensor for cyanogen chloride.

Cyanogen Bromide was procured from Aldrich. A few crystals were placed in a 25 ml erlynmeyer which was fitted with a two hole rubber stopper through which tubing would conduct compressed air in and BrCN vapor out and to the cell containing the Au-C8(11)/MS1302 sensor. BrCN has a 20°C vapor pressure of 82.5 mm Hg and the BrCN vapor and compressed air purges were alternately passed through the cell at a flow rate of ~15 cm³/min.

Initial exposure to the BrCN caused a large increase in the conductivity. It amounted to a 6.5×50,000 = 325,000 Hz signal increase (baseline warning heard). The first purge 3.8×50,000 = 190,000. The second BrCN exposure increased the signal to an off-scale value but the second air purge returned the conductivity (corresponding to that) level to the baseline before this 2nd BrCN exposure. Likewise a third BrCN exposure sent the conductivity level to a value off scale and purge returned it to its previous baseline.



The sensor used was that of Au-C8(11) #2 N74501-38 which was previously characterized by an 50 mV current measurement. Another measurement was made at the new baseline. The conductivity change is as follows:

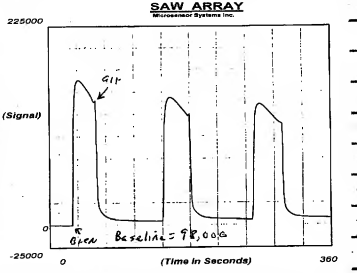
Read and understood (obtain two signatures):

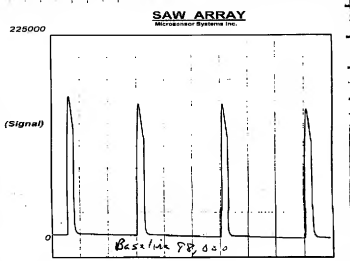
Witness _____ Date _____ Signature Arthur John Date 6-4-97

Witness _____ Date _____

33 nA (50 mV) before BrCN exposure → 250 nA (50 mV) after BrCN exposure

After adjustment of the voltage & resistance of the AC circuit the BrCN response was kept on scale and exposure/response was measured for several cycles each time returning to the same baseline (98,000). The response is extremely fast and strong. Repeated exposures had an apparent decline in signal response intensity. This proved to be more a result of evaporation rate of the BrCN crystal being too slow for the flow rate through the flask. The sensor was allowed to set for 2 hr. Exposures were then made for a 30 sec duration during which the signal follows the exponential dilution in the BrCN flask followed by a six minute purge - allowing greater time for BrCN recovery. The signal in this case did not diminish with repeated exposure.





Signal = 6 × 25000 = 150,000

Signal/baseline = 150,000 / 98,000 = 1.53

$\frac{n_{BrCN}}{n_{air}} = \frac{82.5}{750} = 0.109 = \frac{7.09 \times 10^4}{10^6} \approx 108,000$ ppm

Assuming linear f_n - $\frac{1.53 \text{ fract. of change}}{108,000}$ = 0.000014 fract. change/ppm All 0.014% change/ppm is 100 ppm should produce a 1.4% conductivity change

Read and understood (obtain two signatures):

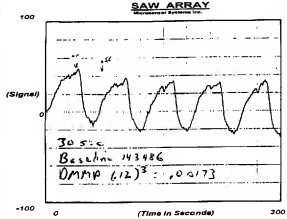
Witness _____ Date _____ Signature [signature] Date 6-4-97

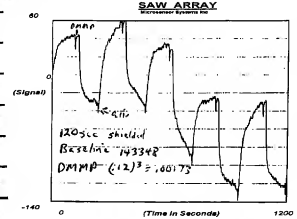
Witness _____ Date _____

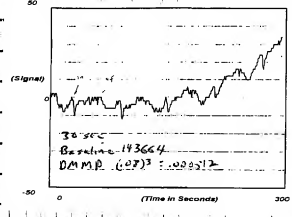
DMMP Response of Au$_{C_8}$ 1:1

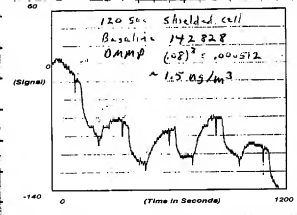
Objective: Determine sensitivity limit of generic Alkanethiol stabilized gold cluster sensor to dimethylmethylphosphonate (DMMP).

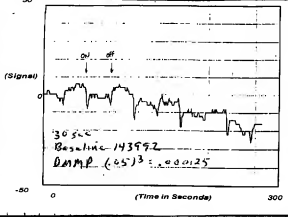
The Au$_{C_8}$ (1:1) / MSI 302 device was mounted in the vapor exposure cell and connected to the vapor generator by a minimum tube length.

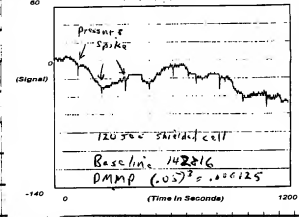












Read and understood (obtain two signatures):

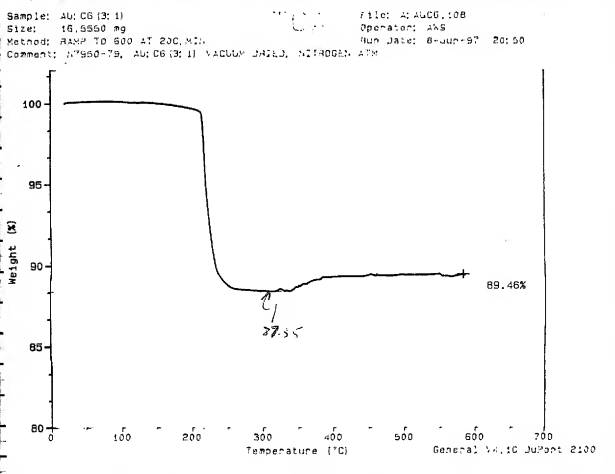
Witness Date Signature Date 6-4-99

Witness Date

TGA of $Au C_6$ (3:1) $Au C_8$ (3:1)
$Au C_2\phi$ (1:1), $Au C_1\phi$ (1:1) and $Au C_1\phi OMe$

TGA's were run at 20°C/min under N_2 on 15 - 20 mg samples

Sample	AuC_6 (3:1)	$Au C_8$ (3:1)	$Au C_2\phi$ (1:1)	$Au C_1\phi$ (1:1)	$Au C_1\phi OMe$
File name	AuC6.108	AuC8.105	$Au C_2 C_6$.101	$Au C_1 C_6$.101	$Au C_1 C_6 OMe$.101
W_{Au}					

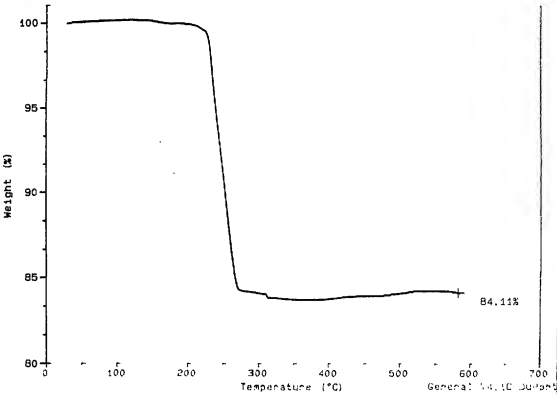


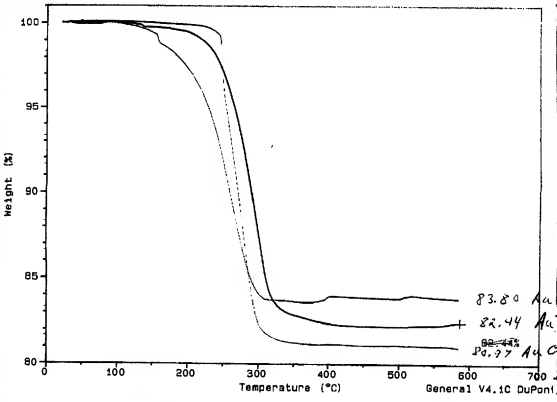
Read and understood (obtain two signatures):

Witness Date Signature Date 6/10/97

Witness Date

Sample: AuCB(3:1) File: A:AUCB.13G
Size: 15.0960 mg Operator: AXS
Method: HAWK TO 600 AT 20C, N2 Run Date: 8-JUN-97 20:06
Comments: 970606-80, AUCB(3:1) VACUUM DRIED, NITROGEN ATM





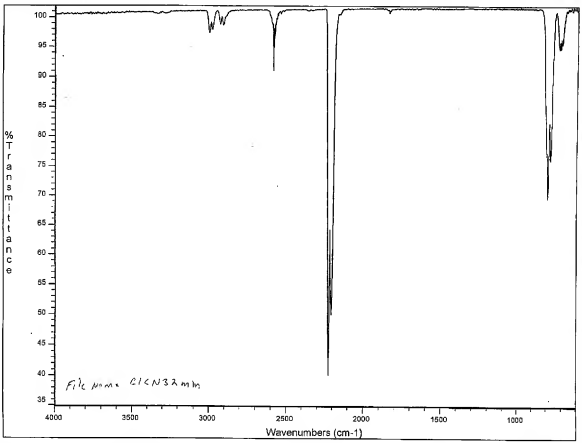
Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 6-10-97

Witness _____ Date _____

Cyanogen Chloride

Cyanogen chloride was condensed in a trap at -78°C from a ClCN cylinder (Matheson) transferred to a vacuum line vacuum distilled (-24°C) and ~~then~~ Transferred to a storage bulb (~700 mm) An IR spectrum (ClCN 32 mm) of the purified vapor was obtained



ead and understood (obtain two signatures):

/itness _____ Date _____ Signature Arthur Shaw Date 6-11-97

/itness _____ Date _____

UV-Vis Characterization of AuC_6 (3:1), $Au C_2$ (3:1), $Au C_2\phi$ (1:1), $Au C_1\phi$ (1:1) and $Au C_1\phi OMe$ (1:1)

(Continuation from NJ 7550-66)

C_6 3:1

1.423 mg / 17.8736 g
0.0799 mg/g
C6 03101
$A_{507} = 1.3383$
$A/b_c = 16.75$

5.2420 g CSI / 10.4045 g
0.4403 mg/g
C6 03102
$A_{507} = 0.6765$
$A/b_c = 16.79$
$a(6) = 11.3 (2510)$

C_2 (3:1)

1.802 mg / 22.5542 g
0.0799 mg/g
C8 03101
$A_{507} = 1.2252$
$A/b_c = 15.33$

4.7837 g CSI / 9.5885 g
0.0399 mg/g
C8 03102
$A_{507} = 0.6183$
$A/b_c = 15.25$
$a(6) = 10.3 (2418)$

$C_2\phi$ (1:1)

1.762 mg / 14.5510 g
0.07986 mg/g
C2C6 0111
$A_{507} = 1.1323$
$A/b_c = 14.18$

3.7484 g CSI / 7.5346 g
0.0397 mg/g
C2C6 0112
$A_{507} = 0.5666$
$A/b_c = 14.27$
$a(6) = 9.64 (2346)$

$C_1\phi$ (1:1)

1.440 mg / 18.0590 g
0.07974 mg/g
C1C6 0111
$A_{507} = 1.0460$
$A/b_c = 13.17$

4.8889 g CSI / 9.8178 g
0.0397 mg/g
C1C6 0112
$A_{507} = 0.5258$
$A/b_c = 13.24$
$a(6) = 8.95 (2100)$

$C_1\phi OMe$ (1:1)

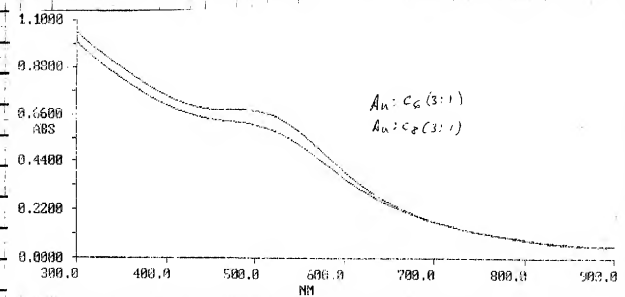
1.749 mg / 21.9119 g
0.07982 mg/g
C1C6 OM01
$A_{507} = 1.1358$
$A/b_c = 14.28$

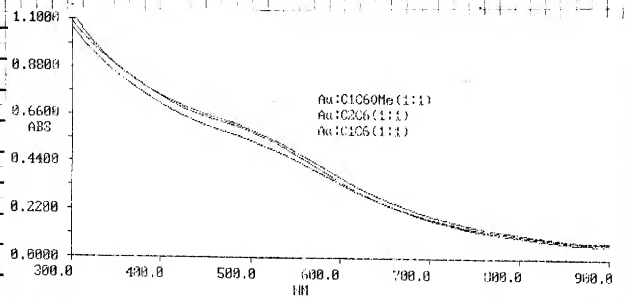
3.8308 g CSI / 7.6113 g
0.04012 mg/g
C1C6 OM02
$A_{507} = 0.5749$
$A/b_c = 14.31$
$a(6) = 9.67 (2310)$

Read and understood (obtain two signatures):

Witness Date Signature Arthur Chu Date 6-11-97

Witness Date





Read and understood (obtain two signatures):

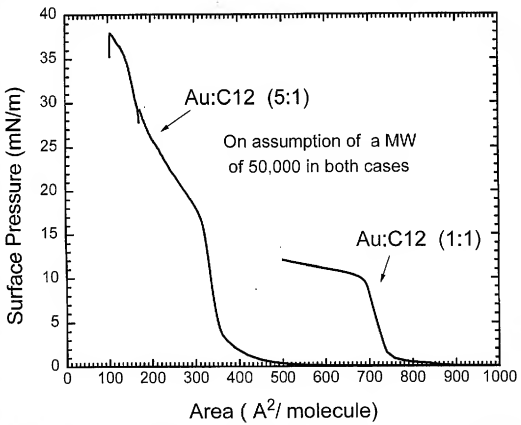
Witness _____ Date _____ Signature Arthur Snow Date 6-11-93

Witness _____ Date _____

Langmuir - Blodgett Isotherm of $Au:C_{12}$ (1:1) and $Au:C_{12}$ (5:1)

Objective: Obtain measures of ligand-shell and void component in packed $Au:C_{12}$ (1:1) and $Au:C_{12}$ (5:1) solids. Also should be able to correlate surface density measurement with bulk density measurement.

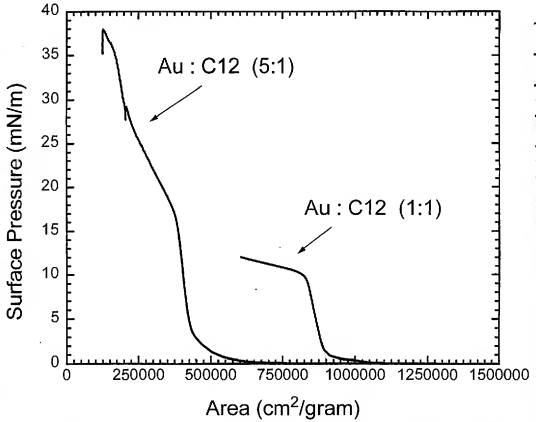
The measurement was conducted by W. R. Barger.



Read and understood (obtain two signatures):

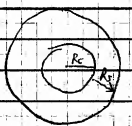
Witness Date Signature Date 6-12-97

Witness Date



The following Analysis was derived to utilize the surface densities from the above data

Surface density $\rho' = \frac{M}{A}$



Mass $M = M_c + M_s$

Core Mass $M_c = 4/3 \pi R_c^3 \rho_c$

Mass Fraction $X_c = \frac{M_c}{M_c + M_s}$ $X_s = \frac{M_s}{M_c + M_s}$ $\Rightarrow M_s = \frac{X_s}{X_c} M_c$

Area $A = A_c + A_s$

$A_c = \pi R_c^2$

$A_s = \pi (R_c + R_s)^2 - \pi R_c^2$ ← this expression estimates A_s is area of shell only

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 6-12-89

Witness _____ Date _____

$$\rho' = \frac{M}{A} = \frac{M_c + M_s}{A_c + A_s} = \frac{M_c + \frac{X_s}{X_c} M_c}{\pi R_c^2 + \pi (R_c + R_s)^2 - \pi R_c^2} = \frac{M_c \left(1 + \frac{X_s}{X_c}\right)}{\pi (R_c + R_s)^2}$$

$$\rho' = \frac{4/3\ \pi R_c^3 \rho_c \left(1 + \frac{1 - X_c}{X_c}\right)}{\pi (R_c + R_s)^2} = \frac{4/3\ R_c^3 \rho_c \frac{1}{X_c}}{(R_c + R_s)^2}$$

$$(R_c + R_s)^2 = \frac{4}{3} R_c^3 \frac{\rho_c}{\rho'} \frac{1}{X_c}$$

$$\boxed{R_s = \left[\frac{4 R_c^3 \rho_c}{3 \rho' X_c}\right]^{1/2} - R_c}$$

Cluster	X_c	R_c	ρ'	ρ_c
$Ad_{C_{12}}(1:1)$	.7549	1.14 nm	19 / 9000000 cm^2	19.3 g/cm^3
$Au_{C_{12}}(5:1)$	.8709	2.97 nm	19 / 45000000 cm^2	19.3 g/cm^3

$$R_{s_{Ad_{C_{12}}(1:1)}} = \left[\frac{4 (1.14\ nm)^3 19.3\ g/cm^3}{3 \left(\frac{19}{9000000\ cm^2}\right) .7549} \times \left(\frac{cm}{10^7 nm}\right)\right]^{1/2} - 1.14 nm$$

$$= \left[2.047\ nm^2\right]^{1/2} - 1.14\ nm = 0.99\ nm$$

$$R_{s_{Au_{C_{12}}(5:1)}} = \left[\frac{4 (2.97\ nm)^3 19.3\ g/cm^3}{3 \left(\frac{19}{45000000\ cm^2}\right) .8709} \left(\frac{cm}{10^7 nm}\right)\right]^{1/2} - 2.97\ nm$$

$$= \left[34.83\ nm^2\right]^{1/2} - 2.97\ nm = 2.93\ nm$$

R_s is not near the same value for the two clusters. Conclusion: Approximating A_s by πR_s^2 is probably not correct. Modified derivation on next page solves for A_s rather than R_s. Also R_s accommodates void area.

Read and understood (obtain two signatures):

Witness Date Signature Date 6-12-97

Witness Date

A_s = non Core area in L-B Film

$$\rho' = \frac{M}{A} = \frac{M_c + M_s}{A_c + A_s} = \frac{M_c + \frac{x_s}{x_c} M_c}{A_c + A_s} = \frac{M_c\left(1 + \frac{x_s}{x_c}\right)}{A_c + A_s}$$

$$A_c + A_s = \frac{M_c}{\rho'}\left(1 + \frac{1 - x_c}{x_c}\right) = \frac{\frac{4}{3}\pi R_c^3 \rho_c}{\rho'} \frac{1}{x_c}$$

$$\boxed{A_s = \frac{4\pi R_c^3 \rho_c}{3\rho' x_c} - \pi R_c^2}$$

$$A_{s_{Au\,C_{12}(1:1)}} = \left[\frac{4\pi\left(\frac{1.14}{2}\text{nm}\right)^3 19.3\ \frac{\text{g}}{\text{cm}^3}}{3\ \frac{19}{900000\ \text{cm}^2} \cdot .7549} \cdot \frac{\text{cm}}{10^7\text{nm}}\right] - \pi\left(\frac{1.14}{2}\text{nm}\right)^2$$

$$= \underset{14.28}{6.45\ \text{nm}^2} - \underset{4.018}{2.32\ \text{nm}^2} = \underset{10.20}{4.1\ \text{nm}^2}$$

$$A_{s_{Au\,C_{12}(5:1)}} = \left[\frac{4\pi\left(2.97\ \text{nm}\right)^3 19.3\ \frac{\text{g}}{\text{cm}^3}}{3\ \frac{19}{456000\ \text{cm}^2} \cdot .8709} \cdot \frac{\text{cm}}{10^7\text{nm}}\right] - \pi(2.97\ \text{nm})^2$$

$$= 109.4\ \text{nm}^2 - 27.7\ \text{nm}^2 = 81.7\ \text{nm}^2$$

	A_s	A_c	$\frac{A_c}{A_c + A_s}$
$Au\,C_{12}(1:1)$	~~16.20~~ 10.20	~~2.5~~ 4.05	~~.36~~ .29
$Au\,C_{12}(5:1)$	81.7	27.7	.25

The $\frac{A_c}{A_c + A_s}$ (fraction of area occupied by core) indicates packing is more efficient for smaller chains

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Arthur Snow Date 6-12-97

Witness _____ Date _____

Density Measurement of AuC_{12} 1:1 and AuC_{12} 5:1

Objective - obtain density to determine ligand shell and void volume in cluster matrix

Samples of AuC_{12} 1:1 and Au_{C12} 5:1 were prepared by depositing cone solute/toluene into 12 mg OSC pans and evaporating and vacuum drying.

AuC_{12} 1:1	Au_{C12} (5:1)
12.8823 mg Pan	12.642 mg Pan
26.6163 mg Pan + C_{12} 1:1	46.693 mg Pan + C_{12} 5:1

A 6 week time lapse occurred between the sample preparation and the measurement below.

A Mettler MT5 (.001 mg sensitivity) balance was used with access to a hangar from below. A platinum wire (very thin) loop in ½ in was placed over the balance hangar rod and a second very thin platinum wire was fashioned with a helical basket at one end and a hook to connect to the loop at the other end as illustrated at right

The balance was zeroed with the loop in place, then the wire basket was connected to the loop, weighed in air, loaded with the sample pan & weighed in air, sample pan removed and basket immersed in water (3X distilled) weighed and sample loaded and immersed in the basket and sample weighed under water.

Data were taken as follows:

Read and understood (obtain two signatures):

Witness Date Signature Date 6/12/97

Witness Date

Blank	Wire	Wire + pan		Wire	Wire + pan	
air	31.91	43.73	±.1	35.95	48.60	±.1
Water	31.72	39.24	±.1	34.83	43.02	±.1

AuC_{12} (1:1)

	Wire	Wire + pan	
air	35.35	62.27	
Water	35.72	53.23	±.1

$AuCa$ (5:1)

	Wire	Wire + pan	
air	35.99	82.26	±.1
Water	35.42	64.50	±.1

Specific Gravity Displacement Ref. ASTM D972-66 p.428

$$Sp\ gr = \frac{W_{air,s}}{W_{air,s} - W_{water,s}}$$

$W_{air,s}$ = wt of sample in air = $W_{air,sample} - W_{air,wire}$

$W_{water,s}$ = wt of sample in water = $W_{water,sample} - W_{water,wire}$

Blank #1 $sp\ gr = \frac{(43.73 - 31.91)}{(43.73 - 31.91) - (39.24 - 31.72)} = \frac{11.82}{4.30} = 2.75$

#4 $sp\ gr = \frac{(48.60 - 35.95)}{(48.60 - 35.95) - (43.02 - 34.83)} = \frac{12.65}{4.48} = 2.84$

for Aluminum literature (CRC) $sp\ gr = 2.70$

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Arthur Chow Date 6-14-67

Witness _____ Date _____

$AuC_{12}(1:1)$ $Sp\,gt = \frac{(62.27-35.35)}{(62.27-35.35)-(53.23-35.72)} = \frac{26.92}{9.41} = 2.86$

$AuC_{12}(5:1)$ $Sp\,gt = \frac{(82.26-35.99)}{(82.26-35.99)-(64.50-35.42)} = \frac{46.27}{17.19} = 2.69$

These results for the AuC_{12} complexes appear to be low but Blank is accurate

Densities for the AuC_{12} complexes are obtained as follows

Component a = Aluminum pan Component b = AuC_{12} complex

$$\rho = \frac{M_a + M_b}{V_a + V_b} \qquad \rho_a = \frac{M_a}{V_a}, \quad \rho_b = \frac{M_b}{V_b}$$

$$\rho = \frac{M_a + M_b}{\frac{M_a}{\rho_a} + \frac{M_b}{\rho_b}}$$

$$\frac{M_a}{\rho_a} + \frac{M_b}{\rho_b} = \frac{M_a + M_b}{\rho}$$

$$\frac{M_b}{\rho_b} = \frac{M_a + M_b}{\rho} - \frac{M_a}{\rho_a}$$

$$\boxed{\frac{1}{\rho_b} = \left(\frac{M_a}{M_b} + 1\right)\frac{1}{\rho} - \frac{M_a}{M_b}\frac{1}{\rho_a}}$$

$AuC_{12}(1:1)$ $M_a = 12.825$ mg $M_b = 26.163 - 12.825 = 13.338$ mg.

$\rho_a = 2.75$ $\rho = 2.86$

$$\frac{1}{\rho_b} = \left(\frac{12.825}{13.338} + 1\right)\frac{1}{2.86} = \frac{12.825}{13.338}\frac{1}{2.75} = 0.336$$

$$\rho_b = 2.97$$

$AuC_{12}(5:1)$ $M_a = 12.642$ $M_b = 46.693 - 12.642 = 34.051$

$\rho_a = 2.75$ $\rho = 2.69$

$$\frac{1}{\rho_b} = \left(\frac{12.642}{34.051} + 1\right)\frac{1}{2.69} = \frac{12.642}{34.051}\frac{1}{2.75} = 0.374$$

$$\rho_b = 2.67$$

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature Arthur Snow Date 6-14-97

Witness _____ Date _____

Thickness Calculation of $Au\,C_{12}(1:1)$ and $Au\,C_{12}(5:1)$

$Au\,C_{12}(1:1)$ $\rho' = \frac{1\,g}{100000\,cm^2}$ (page 34)

$\rho = 2.97\ g/cm^3$

$t = \frac{\rho'}{\rho} = \frac{1g}{100000\,cm^2} \times \frac{1}{2.97\ g/cm^3} \times \frac{10^7\,nm}{cm} = 3.7\,nm$

$Au\,C_{12}(5:1)$ $\rho' = \frac{1g}{450000\,cm^2}$ page 34

$\rho = 2.69\ g/cm^3$

$t = \frac{\rho'}{\rho} = \frac{1g}{450000\,cm^2} \times \frac{1}{2.69\ g/cm^3} \times \frac{10^7\,nm}{cm} = 8.3\,nm$

Comparison with TGA derived result

	t (nm)	$2R_{core}$	$2(R_{core}+R_{shell})$ (page 5)	R_{shell}
$Au\,C_{12}(1:1)$	3.7	2.3	4.0	.88
$Au\,C_{12}(5:1)$	8.3	5.9	8.6	1.35

The agreement between t and $2(R_{core}+R_{shell})$ appears to be very reasonable and perhaps vindicates the apparently low Au C12 density measurements.

Read and understood (obtain two signatures):

Witness Date Signature Date 6-14-57

Witness Date

Au:C$_1\phi$ (1:1) and Au:C$_1\phi$OMe Coated MSI 302 Devices

Objective: Preparation of Au:C$_1\phi$ (1:1) and Au:C$_1\phi$OMe coated devices for toluene, TCE, 1-propanol and N_2O vapor isotherm measurement

Four MSI 302 devices were inspected at 500x, rinsed with CH_2Cl_2 and plasma cleaned 12 hr prior to coatings. Depositions were done in groups of two

Au C$_1$C$_2$ (1:1) N7957-11) 10mg/ml 11.0mg/1.628 g CH_2Cl_2

A 10.0 mg/ml solution was prepared and all brushed onto the pair of cleaned MSI 302 devices mounted and preheated to 120°C 16 one sec passes. Diagnostic current measurement was made at 50 mV

Au C$_1\phi$ (1:1) #1 — 146 μA

Au C$_1\phi$ (1:1) #2 — 577 μA

this compares with a 7.6 μA of Au:C$_2\phi$ (N7957-8)

this appears anomalously large and it was noted that coated brass of heater developed a gold metallic character. Heating had been continued ~5 min at the deposition - this high number might be the result of a thermal instability

Au C$_1$C$_6$ (1:1) N7957-15) 11.2 mg/1.658 g CH_2Cl_2

A 10.0 mg/ml solution was prepared and all brushed onto a pair of cleaned MSI 302 devices preheated to 120°C. The application was 16 one sec passes. A diagnostic current measurement was made at 50 mV

Au C$_1$C$_6$OMe (1:1) #1 — 19.2 μA

Au C$_1$C$_6$OMe (1:1) #2 — 16.2 μA

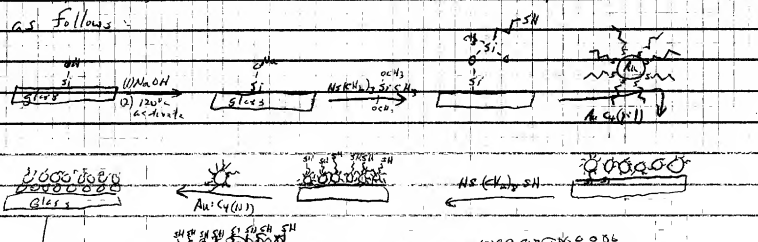
Read and understood (obtain two signatures):

Witness Date Signature Arthur Date 6-15-87

Witness Date

Self-Assembly of $Au:C_n(X:Y)$ / $C_8(SH)_2$ on Glass

Objective: Demonstration that the $Au:C_n(X:Y)$ cluster system can be deposited as uniform films of controlled thickness by a self-assembly technique. Further, these films are stabilized to solvents by intercluster linkages using the difunctional $HS(CH_2)_8SH$. Steps are illustrated as follows:



This concept is also illustrated on N7942-9

The procedure is

① A Fisher Plane microscope slide was cut into a 1 x ½ inch rectangular piece.

② It was washed in $CHCl_3$ (30 min), 5% NaOH (10 min), 3x distilled water.

③ Activated by 2 hr at 120°C

④ Immersed in a 0.061 g $HS(CH_2)_2CH_2C_6H_3(OCH_3)CH_3(OCH_3)$ / 0.683 g Hexanes (HPLC) for 13 hr

Read and understood (obtain two signatures):

Witness _____ Date _____ Signature _____ Date 6-20-97

Witness _____ Date _____

⑤ Wash in hexanes 1 min (2 strokes of washing)

⑥ Immerse in 1.6 mg $Au(C_4H_8)Cl$ / 2.21 g $CHCl_3$ solution 5 min

⑦ Wash 1 min in hexanes sequential dips in two containers

⑧ Immerse in 6.8 mg $NS(CH_3)_2SH$ / 2.20 g $CHCl_3$ 5 min

⑨ Repeat steps ⑤ → ⑧ four times to get a 5 layer film.

Each subsequent dip effected effected a darkening intensity of the film (from a faint tinge → an easily perceived dark purple) The film dekored in the glass after washing 20 min with fresh $CHCl_3$. Microscope observation revealed regular features which also appeared on a control blank subjected to cutting treatment. - coloration appeared uniform.

Read and understood (obtain two signatures):

Witness Date Signature Arthur Ko Date 6-29-93

Witness Date